World Cotton Research Conference-4, Lubbock, Texas, U.S.A.

Fiber Quality Evaluation and Preservation

Centre de Coopération Internationale en Recherche Agronomique pour le Développement

Calibration, measurements stability and replacement of standard cottons for an FMT3

Lubbock 2007, Sept 10-14.

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Outline of the presentation

- Introduction
- Observational study:

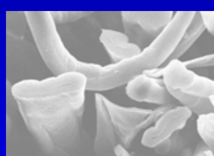
  Check of the validity of calibration methods
- Experiment:

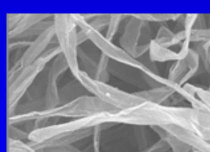
  Evaluation of new reference material
- Discussion-Conclusions

Introduction

How is maturity evaluated ?

Directly with a microscope, or indirectly via resistance to air flow





English SDL-Atlas maturimeters
FMT 1, FMT1A, 1B,
FMT 2, FMT3, micromat.

Too long not Appropriate For many samples

Only Mike 2 x 3,24g Raw fiber

Introduction

All these parameters are calculated from only two depressions:

PL (low pressure) and PH (high pressure)

IM : micronaire   MR : Maturity Ratio

PM% : Percent maturity fiber

H : Fineness (mtex)   Hs : Fineness Standard (mtex)

The formulas are the following:

$$IM = \frac{850}{PL + 40} + 0,6$$

$$H = \frac{60000}{PL} \times \frac{PH}{PL}^{1,75}$$

$$MR = 0,247*(PL)^{0,125} \times (PL/PH)^{2}$$

$$PM = 100 * (MR - 0,2) * (1,565 - 0,471\ MR)$$

Introduction

Facts about Standards and 2 calibration questions

- FMT3 calibration method is with two standards only
- Supplied standard is few, and built-in calibration software is bounded with it
- USDA does NOT provide standards for maturity
- USDA micronaire standards can be used instead, one has to replace the calibration method as well

1/ what calibration method is valid ?

Introduction

Facts about Standards and 2 calibration questions

USDA standards eventually come out of stock

2/ how to evaluate a new reference material to replace them

= CREATE ONE'S OWN <u>REFERENCE MATERIAL</u> !

Introduction

Material and methods

Operating conditions

- **conditioning room at ISO 139 values**
- **dry air pressure of 6 bars.**
- **adjusting one rotameter for the 1 and 4l/mn for the two depressions.**
- **10 or 12 grams of blended and conditioned fiber.**
- **accurate weighing**
- **verifying the values of the SDL values (small quantity).**
- **good preventive maintenance (O-ring).**

Introduction

Large range (PL / PH) of three "old" reference cottons : After 2000

Designation	1301 M 1	0338 C 38	1201 L 1
P L	320.87	241.88	176.18
P H	272.07	193.34	136.71
IM	2.96	3.62	4.53
MR	0.71	0.77	0.78
PM	62.4	68.3	69.7
H	140	168	218
HS	198	218	279

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Checking the calibration methods

Introduction

The observed and corrected standards values are stored in a data base SISTER ® (Gourlot & Giner, 99).

To verify that the analysis are stable in time, we have studied 1445 data triplets of these three cottons tested during 6 years by 12 technicians for each lot of samples.

Two different corrections are studied:

- **multiplicative factor**
- **linear regression**

Checking the calibration methods

Statistical analysis*

Validity of the correction factor

A multiplicative model is a regression model without an intercept : for cotton *i* and day *j*, $Y_{ijt} = a_{it}\, x_j + b_i$

For a correction factor to be valid, regression equations should have a nil intercept, on the average.

In practice, the estimated intercepts that can be obtained at each calibration operation, should then be distributed randomly around 0.

Student's test (*) of zero mean of the intercepts

*** : with SAS/Stat & SAS/Graph, v9**

Checking the calibration methods

Statistical analysis*

Validity of the correction by linear regression

Linear model $$E[Y_{ijt}] = a_{jt} + b_{jt}x_i + c_i + d_{ij}$$

where **I = cotton** **j= technician**
t = time **xi = known cotton value**
Yijt = measured value
= regression for one day, one tech

$Y_{ijt} = a_{jt} + b_{jt}x_i \;\; c_i + d_{ij}$ **= departures from linearity**

ANOVA, Fisher's test (*) of departure from linearity

*** : SAS/Stat (GLM), v9.**

Checking the calibration methods

Results and discussion

Stability and variability in time before and after correction: graphic description*

We show :
a large dispersion of the raw values of PL and PH (a)

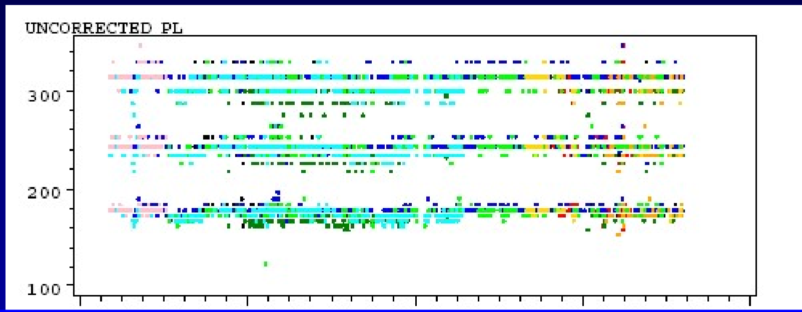
The rounded values of PL have some points alignments :
Is it a impact on the accuracy?

PH was not visibly affected.

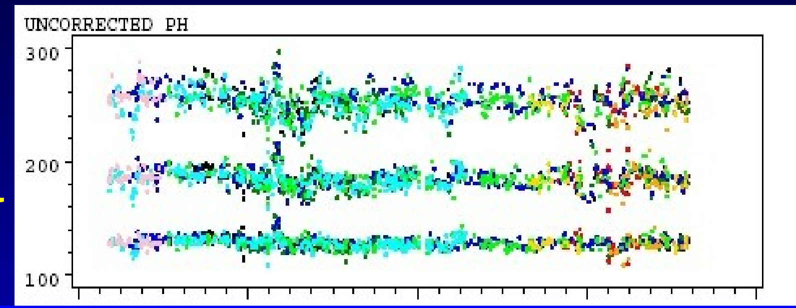
The simple correction factor stabilizes the measurements (b)

Improvement can be achieved with correction by regression (c)

Checking the calibration methods



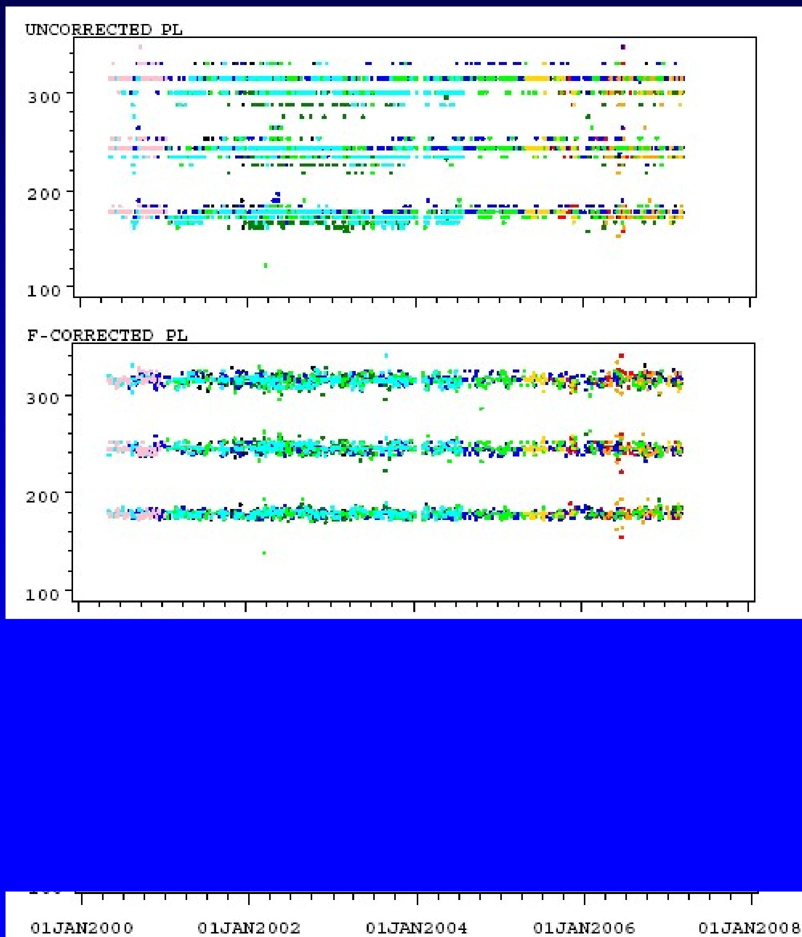
a



PL

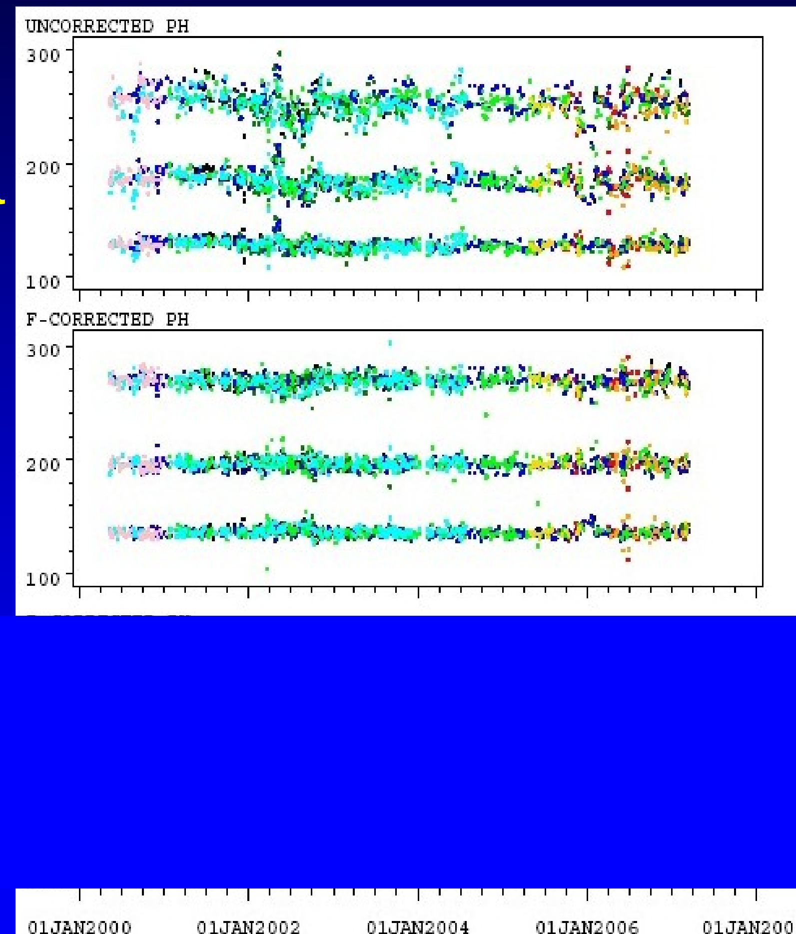
PH

Checking the calibration methods



a

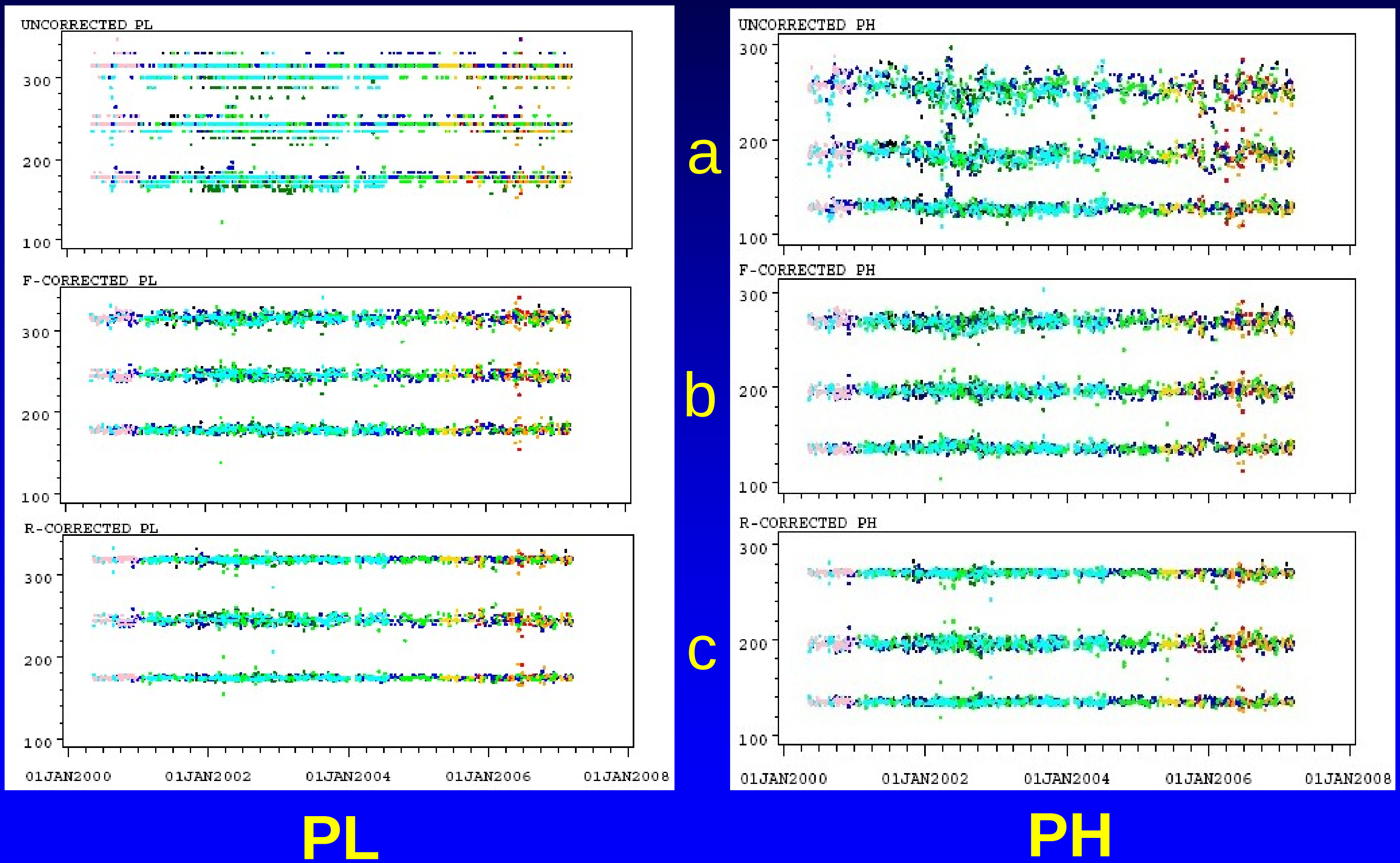
b



PL

PH

Checking the calibration methods



Checking the calibration methods

Results and discussion

Long term tendencies of the raw values

Depression	Cottons	Equations	P value of test of $\lvert slope\rvert \neq 0$
PL	L01	$PLb = +84_*10^{-06}X + 175.3$	0.4984
	C38	$PLb = -116_*10^{-06}X + 241.4$	0.4775
	M01	$PLb = -205_*10^{-06}X + 310.0$	0.3748
PH	L01	$PHb = -585_*10^{-06}X + 129.6$	<0.0001
	C38	$PHb = -588_*10^{-06}X + 186.1$	0.0003
	M01	$PHb = -973_*10^{-06}X + 255.2$	<0.0001

Table 3

Validity of the correction factor method

The average of the intercepts are all significantly different from zero for each technician with only one exception = bias

		Dependent variable					
		PL			PH		
Operator	n	Mean	Std Error	t	Mean	Std Error	t
1	80	13.63	1.28	10.65	5.88	1.49	3.95
2	56	14.45	1.96	7.37	4.38	1.86	2.35
3	168	17.77	0.92	19.32	7.08	0.89	7.96
4	408	12.37	0.48	25.77	2.17	0.48	4.52
5	37	16.74	2.09	8.01	7.44	1.82	4.09
6	260	14.77	0.87	16.98	4.76	0.82	5.80
7	51	15.81	1.39	11.37	5.24	1.21	4.33
8	237	16.03	0.76	21.09	6.50	0.7	9.29
9	56	9.87	1.45	6.81	-1.85	1.48	-1.25

Table 4

Checking the calibration methods

Results and discussion

Validity of correction by regression

- non-linearity test is significant
=> relation between observed and theoretical values is not linear.

Depression	Source	DF	Type I SS	Mean Square	F Value	Pr > F
PL	NOMSTD	1	20348.25	20348.25	658.53	<.0001
	TECH*NOMSTD	8	732.90	91.61	2.96	0.0026
PH	NOMSTD	1	16836.05	16836.05	428.42	<.0001
	TECH*NOMSTD	8	694.81	86.85	2.21	0.0240

Table 5

Checking the calibration methods

Results and discussion

Validity of correction by multiplicative factor and by linear regression

In theory, neither correction by multiplicative factor nor that by regression is satisfactory.
However, the comparison shows that these deviations are not of great practical importance.

Cottons (Nb)	Theoretical		Observed		F-Corrected		R-Corrected	
	PL	PH	PL	PH	PL	PH	PL	PH
L01 (1442)	176.2	136.7	175.5	128.3	178.6	136.5	174.7	135.2
C38 (1454)	241.9	193.3	241.1	184.8	245.3	196.5	245.4	196.6
M01 (1440)	320.9	272,1	309.6	253.1	314.9	269.0	318.9	270.3
Mean (4338)	246.3	200.7	242.1	188.7	246.3	200.6	246.3	200.7

Table 6

Checking the calibration methods

Conclusion:

Validity of correction by multiplicative factor and by linear regression

The correction methods stabilize the results and do not induce noticeable bias

Either can be used to evaluate a new reference material, to renew the finishing standard.

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Evaluation of new reference material

Cottons and technicians

Two new reference material where evaluated with 7 technicians and the following experimental design:

- For C39 evaluation one rep and 25 rolls.

Roll number	Operators						
	1	2	3	4	5	6	7
01, 04, 09, 15, 25	X	X					X
06, 10, 13, 19, 24	X		X				X
03, 08, 11, 18, 20	X			X			X
05, 12, 14, 17, 23	X				X		X
02, 07, 16, 21, 22	X					X	X

Table 1

Evaluation of new reference material

Cottons and technicians

- For L02 evaluation two replications and 20 rolls

	Replicate													
	A							B						
	Operators							Operators						
Roll number	1	2	3	4	5	6	7	1	2	3	4	5	6	7
01, 04, 09, 15	X	X					X	X	X					X
02, 07, 16, 20	X		X				X	X		X				X
03, 08, 11, 18	X			X			X	X			X			X
05, 12, 14, 17	X				X		X	X				X		X
06, 10, 13, 19	X					X	X	X					X	X

Table 2

Evaluation of new reference material

Material and methods

Validity of the calibration by correction factor

for cotton i and technician j

$$E(Y_{ijk}) = \mu \alpha_i \beta_k$$

on the log scale, linear model without an interaction term

$$Log(E(Y_{ijk})) = m + a_i + b_k$$

Gaussian errors in the natural scale or no longer gaussian nor of constant variance on the log scale

$\Rightarrow$ need for a generalized linear model

(Mc Cullagh and Nelder, 83)

*** : SAS/Stat GENMOD procedure & Glimmix macro**

Evaluation of new reference material

Material and methods

Validity of the calibration by correction factor

In the LOG scale, the cotton x technician interaction is significant in the L02 experiment, but not in the C39 experiment.

Type 3 Tests of operator x cotton interaction

		Num DF	Den DDL	F Value	Pr > F
PL	C39	18	37.6	1.64	0.0990
	L02	18	119.0	3.59	<.0001
PH	C39	18	44.6	1.08	0.3981
	L02	18	121.0	3.77	<.0001

Table 7

Evaluation of new reference material

Results and discussion

Validity of the correction by linear regression :.

The average deviation by cotton (cotton effect) and the variations in these deviations according to technician (tech*cotton interaction) are significant only for PL in experiment L02.

Cotton	Depression	Tested effect	DF	Type I SS	Mean Square	F Value	Pr > F
C39	PL	departure	1	29.94	29.94	0.69	0.4277
	PH	departure	1	62.78	62.78	1.02	0.3391
L02	PL	departure	1	123.41	123.41	5.43	0.0366
		Tech. x dep.	6	93.86	15.64	0.69	0.6632
	PH	departure	1	59.42	59.42	3.07	0.1035
		Tech. x dep.	6	164.68	27.45	1.42	0.2805

Table 8

Evaluation of new reference material

Results and discussion

Estimated values of PL and PH of the reference material

The newest reference for the lab are the following.
The results corrected by multiplicative factor have a smaller standard error than those corrected by regression, and the biases of both methods are small

Cotton	Depression	F-Corrected		R-Corrected	
		Mean	Precision	Mean	Precision
C39	PL	274.66		277.07	
	PH	220.95		221.57	
L02	PL	183.28	0.79	177.60	1.22
	PH	129.26	0.82	127.06	1.33

Table 9

Evaluation of new reference material

Impact of the PL and PH errors on the derived parameters

	PL	PH	Error	IM	Mat.	PM%	Fin H	Fin HS
L02	181,73	127,65	-1,55 / -1,61	4,43	0,96	84,5	177,9	185,5
	183,28	129,26	0	4,41	0,95	84,0	177,7	186,5
	184,83	130,87	+1,55 / +1,61	4,38	0,95	83,5	177,4	187,5
Variation			Same way	0,03	0,0065	0,505	0,26	1,25
			Cross way	0,03	0,04	3,11	8,01	16,42

Evaluation of new reference material

Results and discussion

Theoretical reference values of the two new cottons:

Before june 2007

Designation	1301 M 01	0338 C 38	1201 L 01
P L	320.87	241.88	176.18
P H	272.07	193.34	136.71
IM	2.96	3.62	4.53
MR	0.71	0.77	0.78
PM	62.4	68.3	69.7
H	140	168	218
HS	198	218	279

After june 2007

1301 M 01	0339 C 39	1202 L 02
320.87	274.66	183.28
272.07	220.95	129.26
2.96	3.30	4.41
0.707	0.77	0.95
62.44	68.5	84.0
140	149	178
198	194	186

Evaluation of new reference material

Conclusion:

Evaluation of the reference material in the future

One should avoid counfounding technicians and rolls effects, while preserving feasability

	Replicate														
	A							B							
	Operators							Operators							
Roll number	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**Roll number**
01, 08, 15	**X**							**X**							**14, 18, 20**
02, 09, 16		**X**							**X**						**10, 12, 21**
03, 10, 17			**X**							**X**					**11, 16, 17**
04, 11, 18				**X**							**X**				**06, 07, 15**
05, 12, 19					**X**							**X**			**01, 05, 19**
06, 13, 20						**X**							**X**		**04, 09, 13**
07, 14, 21							**X**							**X**	**02, 03, 08**

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Discussion-Conclusions

Measurements using FMT3 apparatus require calibration.

As calibration gives stable measurements in time, establishment of new reference material maturity values can be set in reference to former cotton reference values.

Calibration software should be modified so that standards can be changed if they are exhausted.

Discussion-Conclusions

It would be preferable to base calibration on three cottons rather than two, with a warning given in case of a sizeable deviation from linearity.

A shift, even limited, is inevitable when a change is made as zero error cannot be guaranteed at a change in reference material.

One overlapping between the successive standards is actually tested at the lab to pass from the old to the newest without shift.

A more adapted experimental design is planned.

Discussion-Conclusions

Find a triplets with both large range for depression **AND** derived characteristics (IM, MR…)

It would also allow an empirical comparison of calibration accuracy by constant factor and by regression.

The Micromat® operates using the same principle, it could be tested in a similar experiment.

Inter-laboratory maturity tests are therefore required so that laboratories can calibrate between each other and avoid individual long-term drift.

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Thank you for your attention!